

#### 4. Improvement of Instrumental Performance in the Presence of Drift

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**Objective:** To demonstrate the broader applicability of the Salit-Turk drift correction procedure for improving the precision of analytical measurements.

**Problem:** Low-frequency noise—often termed drift—results in a poor signal-to-noise ratio for repeated measurements of a given sample and is a common problem for analytical instruments. In 1998, Salit and Turk published an innovative strategy to overcome drift that used all the data in an experiment to create a model of the instrument drift response function. This new approach gave more than 10-fold improvements for precision in ICP-OES measurements. The authors speculated that the method would have general utility for precision chemical metrology and this speculation has now been verified in our laboratory using several analytical techniques, including glow discharge optical emission spectrometry (GD-OES) and atomic absorption spectrometry (AAS).

**Approach:** Drift correction methods that are usually used in GD-OES assume that calibration (and recalibration) can be done in a time frame that is short enough to be unaffected by drift. However, rigorous calibration in GD-OES—spanning a range of calibrants, repetitively measured—is time intensive. Drift does occur, compromising the validity of the calibration and possibly introducing analytical bias into the process. One type of drift that is observed in GD-OES is a burn-to-burn (sample-to-sample) variation of instrumental sensitivity and background. This drift can be caused by reduction of light transmission by the window that separates the glow discharge cell from the vacuum spectrometer.

The method's effectiveness for compensation of drift-induced calibration bias was tested by the determination of C, P, S, and Si in grey cast iron, using 17 CRMs as calibration samples. This analysis accentuated the problem—because of the time per sample and intense conditions required, the vacuum wavelengths measured, and the number of calibrants included.

The drift-corrected, carbon calibration data are graphed above. The well-defined calibration line illustrates the significant improvement that was achieved.

Experiments demonstrating the application of the Salit-Turk drift correction procedure in AAS, both flame and cold vapor, also have been done. In flame AAS, Cu, Fe, and Zn were determined in SRM 909 Human Serum using a dynamic dilution sample introduction pump system and internal normalization with Mn. Precision improved by factors between 3-6 for the elements measured. The procedure was applied in cold vapor AAS determination of Hg in Dust, SRM 2584. A drift of about 13% was observed over 5 hours of data collection, yet the relative precision for each sample ranged from 1-4% (1s). The Hg concentration determined in this material was 5.25 ppm (mg/kg) with a between sample relative standard deviation of 1.7%.

**Results and Future Plans:** The application of the drift correction methodology has improved the quality of NIST measurements, as noted above. The larger impact that can be achieved by its adoption and use outside NIST is also being promoted. Significant progress has been made in its transfer to the broader analytical community. Joint experiments have been done with both primary and secondary reference material providers in the U.S. and abroad; a successful collaboration has been completed with another agency of the U.S. government; and, several co-operative arrangements, both formal and informal, have been developed with analytical instrument manufacturers.

